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Progress has been made in understanding growth patterns of Thin Fine Grain Diamond CVD Films. In the study which was performed by High Resolution Electron Microscopy attention was focused on the fault structure as a function of the position in the grain as well as on the formation of mismatch boundaries within the growing diamond crystal. These boundaries are formed due to mismatch between certain twins within the growing crystal. It was found that these boundaries can serve as precise indicators for the local growth direction of the crystal and thus to lead back to the nucleation point in a given TEM cross section. In an example given in this report the local nucleation point is traced back to a 5-fold twin site.

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TECHNICAL REPORT No. 2

TRANSMISSION ELECTRON MICRSCOPY OF THE CVD DIAMOND FILM/SUBSTRATE INTERFACE

by

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Technical Report Prepared for NIST

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Gaithersburg, MD 20899

and

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March 21, 1991

INTRODUCTION

THIS REPORT INCLUDES RESEARCH RESULTS OBTAINED DURING THE PERIOD JULY 1ST TO SEPTEMBER 30, 1990 WHICH IS THE THIRD QUARTER OF THE FIRST YEAR OF THIS PROGRAM.

PROGRESS HAS BEEN MADE IN UNDERSTANDING GROWTH PATTERNS OF THIN FINE GRAIN DIAMOND CVD FILMS. IN THE STUDY WHICH WAS PERFORMED BY HIGH RESOLUTION ELECTRON MICROSCOPY ATTENTION WAS FOCUSED ON THE FAULT STRUCTURE AS A FUNCTION OF THE POSITION IN THE GRAIN AS WELL AS ON THE FORMATION OF MISMATCH BOUNDARIES WITHIN THE GROWING DIAMOND CRYSTAL. THESE BOUNDARIES ARE FORMED DUE TO MISMATCH BETWEEN CERTAIN TWINS WITHIN THE GROWING CRYSTAL. IT WAS FOUND THAT THESE BOUNDARIES CAN SERVE AS PRECISE INDICATORS FOR THE LOCAL GROWTH DIRECTION OF THE CRYSTAL AND THUS TO LEAD BACK TO THE NUCLEATION POINT IN A GIVEN TEM CROSS SECTION. IN AN EXAMPLE GIVEN IN THIS REPORT THE LOCAL NUCLEATION POINT IS TRACED BACK TO A 5-FOLD TWIN SITE.

THE ROLL OF TWINS IN THE GROWTH OF DIAMOND CVD CRYSTALS HAVE ALSO BEEN INVESTIGATED. IT WAS FOUND THAT TWINS SERVE A VITAL NUCLEATION SITES FOR NEW PLANES AND THAT RAPID GROWTH OF CERTAIN CVD DIAMOND PATTERNS DEPENDS HIGHLY ON TWINING.

THE HIGH RESOLUTION ELECTRON MICROGRAPHS PRESENTED IN THIS REPORT WERE TAKEN ON A JEOL 400 OF OXFORD, ENGLAND.

THE HELP OF DR. J.L.HUTCHISON, THE HEAD OF THE HREM LABORATORY IS GREATLY APPRECIATED.

SPECIMEN PREPARATION

THE DEPOSITION OF FREE-STANDING CVD DIAMOND WAS PERFORMED AT NIST. A SILICON WAFER 3" IN DIAMETER AND 400 MICRONS THICK WAS POLISHED ON ONE SIDE AND THEN CUT INTO SQUARE SUBSTRATES 1.9X1.9 CM EACH. AN INDENTATION 0.95 CM IN DIAMETER WAS GROUND INTO THE CENTER OF THE UNPOLISHED SIDE OF EACH SUBSTRATE. THE DEPTH OF THESE INDENTATIONS IN ROUGHLY HALF THE THICKNESS OF THE WAFER. THE POLISHED SIDE OF THE SUBSTATES WAS THE MANUALLY POLISHED WITH 1 MICRON DuPont DIAMOND POWDER. THE MANUAL POLISH IS NECESSARY TO INCREASE THE NUCLEATION DENSITY AND THUS DEPOSIT DIAMOND FILMS WITH A SMALLER GRAIN SIZE.

THE PROCEDURE FOR THE MANUAL POLISHING IS AS FOLLOWS: DIAMOND POWDER IS SPRINKLED ONTO A LARGE GLASS PLATE AND THEN PRESSED INTO A "PANCAKE BETWEEN TWO GLASS PLATES.THE SILICON SUBSTRATE IS PLACED, POLISHED SIDE DOWN, ON THE DIAMOND POWDER "PANCAKE" AND RUBBED MANUALLY FOR SEVERAL MINUTES IN A CIRCULAR MOTION. IF THE DIAMOND "PANCAKE" BREAKS APART IT MUST BE PRESSED AGAIN. FUSSED SILICA OR OTHER OPTICAL GLASSES CAN BE ALSO PREPARED FOR DIAMOND DEPOSITION THIS WAY. IT IS INTERESTING TO NOTE THAT THE USE OF FINER DIAMOND POWDER SUCH AS 0.125 MICRON HAS LITTLE EFFECT ON THE DIAMOND NUCLEATION DENSITY.

DIAMOND FILMS WERE GROWN ON THE PREPARED SILICON SUBSTRATES IN A MICROWAVE-PLASMA ASSISTED CHEMICAL VAPOR DEPOSITION (MPACVD) SYSTEM.

DEPOSITION CONDITIONS WERE AS FOLLOWS:

MICROWAVE POWER - 1KW

GRAPHITE SUSCEPTOR TEMPERATURE - 650°C

(THE TEMPERATURE OF THE GROWING FILM IS KNOWN TO BE CONSIDERABLY HIGHER AS A RESULT OF THE HEATING IN THE MICROWAVE PLASMA).

GAS PRESSURE - 50 TORR

GAS FLOW RATE - 260 STANDARD CM^3/MIN;

GAS COMPOSITION - 99.5% H_2 , 0.5% CH_4

DEPOSITION TIME - 45 MIN FOR SPECIMEN LR-1 (231C)
120 MIN FOR SPECIMEN LR-2 (222C)

GROWTH RATE - 0.4 MICRONS/HOUR

TOTAL THICK NESS - 0.3 MICRONS FOR SPECIMEN LR-1
0.8 MICRONS FOR SPECIMEN LR-2

OPTICAL REFLECTANCE MEASUREMENTS SUGGEST THAT THE ROOT MEAN SQUARE SURFACE ROUGHNESS OF THESE FILMS IS APPROXIMATELY 0.02 MICRONS.

FOLLOWING DEPOSITION THE SUBSTRATE WAS ETCHED USING STANDARD SOLUTION (SEE REPORT NUMBER 1) AND SQUARES, 3 MM ON THE SIDE WERE CUT FROM THE FREE STANDING FILM. THE DIAMOND SQUARES WERE THEN PLACED BETWEEN GRIDS AND THINNED BY ION MILLING.

THE DEPOSITION RECORDS ARE GIVEN IN APPENDIX #1.

MULTIPLE TWINS AND MISFIT BOUNDARIES

DIAMOND GRAINS WHICH CONTAIN MULTIPLE TWINS HAVE BEEN SEEN IN DIAMOND CUBIC MATERIALS. SEVERAL EXAMPLES INCLUDE SILICON (REF 1,2) AND DIAMOND (REF 3). IN MANY CASES THE DIAMOND PARTICLES POSSESS AN ICOSAHEDRAL SHAPE AND NUMEROUS SUCH EXAMPLES CAN BE OBSERVED BY SCANNING ELECTRON MICROSCOPY. HOWEVER, THE FORMATION OF 5 TWINS WHICH TOUCH AT A POINT IN THE PLANAR ARRANGEMENT AND ALONG A LINE IN THE [110] DIRECTION IN SPACE CAN BE A LOCAL PHENOMENON WHICH DOES NOT CREATE AN ICOSAHEDRALLY SHAPED CRYSTAL SINCE OTHER TWINS DIVERT THE GROWTH DIRECTION DURING THE CONTINUING GROWTH OF THE CRYSTAL.

ONE SUCH EXAMPLE IS SHOWN IN FIGURE 1. IN THIS CASE FIVE TWINS MEET AT A POINT AND THE FORMATION OF THE 7.5° MISFIT BOUNDARY IS CLEAR, AS ILLUSTRATED IN THE INSERT.
SUCH A MISFIT BOUNDARY CAN STRETCH TO THE SURFACE OF THE CRYSTAL OR BE CORRECTED LOCALLY AT ANOTHER TWIN BOUNDARY AS SHOWN IN FIGURE 2. IN ALL CASES THE EDGES OF THE MISFIT BOUNDARY WILL EITHER END AT A TWIN BOUNDARY OR AT THE SURFACE OF THE CRYSTAL.

EVEN THOUGH MISFIT BOUNDARIES ARE ALWAYS FORMED WHEN 5 TWINS MEET AT A POINT, SUCH A MISFIT BOUNDARY CAN FORM ALSO BY A FORMATION OF A SMALLER NUMBER OF TWINS SUCH AS IN FIGURE 3. IN THIS CASE THE MISMATCH BOUNDARY FORMS BETWEEN TWO TWINS AND EXTENDS FROM ONE TRIPLE POINT TO ANOTHER.

MISFIT BOUNDARIES ARE USUALLY FOUND IN THE PERIPHERY OF THE CRYSTAL WHERE THE NUMBER OF TWINS AND THEIR DENSITY IS HIGH (FIGURE 4). A MORE DETAILED ANALYSIS OF MISFIT BOUNDARIES IN CVD DIAMOND WILL BE GIVEN IN ONE OF THE NEXT REPORTS.

MISFIT BOUNDARIES ARE ESSENTIAL TO THE UNDERSTANDING OF THE DIAMOND CVD CRYSTAL GROWTH. THIS IS DUE TO THE FACT THAT THEY ARE THE LOCUS OF THE POINTS OF INTERSECTION OF GROWING PLANES FROM TWO ADJACENT NON MATCHING TWINS. THEY ARE ALIGHNED THEREFORE ALONG THE LOCAL GROWTH DIRECTION OF THE CRYSTAL AND ALLOW THE DETERMINATION OF THE GROWTH DIRECTION AT VARIOUS POINTS OF THE CRYSTAL AS WELL AS THE NUCLEATION POINT OF GROWING PLANES IN THE TEM CROSS SECTION.

AN EXAMPLE OF SUCH A DETERMINATION IS GIVEN IN FIGURE 5. A COMPLETE CROSS SECTION OF A CRYSTAL IS SHOWN AND THE GROWTH DIRECTIONS ARE MARKED BY ARROWS ALONG MISMATCH BOUNDARIES. THE NUCLEATION POINT OF THE GROWING PLANES IN THIS CROSS SECTION CAN BE TRACED BACK TO THE 5-FOLD TWIN SITE (MARKED BY A CIRCLE). THE IMPORTANCE OF THE 5-POINT TWIN SITE TO THE NUCLEATION AND GROWTH OF CVD DIAMOND CRYSTALS WILL BE DISCUSSED LATER IN THIS REPORT.

THE ROLL OF TWINING IN THE GROWTH OF CVD DIAMOND

A LARGE NUMBER OF DIAMOND GRAINS HAVE BEEN INVESTIGATED IN THE COURSE OF OUR STUDY. IN ALL CASES THE GRAINS WERE TWINNED AND THE DISTRIBUTION OF THE TWINS WAS IRREGULAR. SINCE EVEN THE SMALLEST REGIONS CONTAIN TWINS AND MULTIPLE TWINING IS VERY ABUNDANT IT SEEMS THAT THE ROLL OF TWINING IS IMPORTANT IN THE GROWTE OF CVD DIAMOND CRYSTALS

IN GENERAL THE DENSITY OF TWINS WAS FOUND TO BE HIGHER CLOSE TO THE GRAIN BOUNDARIES (SEE FIGURE 5).
IT HAS ALSO BEEN OBSERVED THAT REGIONS CLOSE TO THE EDGE OF THE CRYSTAL WHICH DO NOT CONTAIN A HIGH DENSITY OF TWINS DO NOT GROW AS FAST AS REGIONS WHICH DO. CONSEQUENTLY, REGIONS LEAN IN TWINS LOSE IN THE COMPETITION FOR SPACE TO THE GROWING NEIGHBORING GRAINS AND TEND TO FORM A BAY SHAPE IN THE CROSS SECTION (SEE FIGURE 6).

THE IMPORTANCE OF TWINS TO THE GROWTH OF GERMANIUM HAVE BEEN STUDIED IN THE PAST AND CONSEQUENTLY UTILIZED FOR THE GROWTH OF GERMANIUM DENDRITES FROM THE MELT [REFS 5, 6, 7, 8, 9].

THE TWIN BOUNDARY REENTRANCE ANGLE MODEL OF HAMILTON AND SEIDENSTICKER [REF. 7] HAS SHED LIGHT ON THE VITAL IMPORTANCE OF TWINING IN THE GROWTH PROCESS OF GERMANIUM. THE MODEL FOR FAST GROWTH (ILLUSTRATED IN FIGURE 7) CALLS FOR A PAIR OF PARALLEL TWIN BOUNDARIES WHICH FORM A NEW REENTRANCE ANGLE AS ONE TWIN BOUNDARY CEASES TO SERVE AS A PREFERRED GROWTH SITE. IN THE CASE THAT ONLY ONE TWIN BOUNDARY IS PRESENT A BICRYSTALLINE TRIGONAL SOLID IS FORMED PROVIDED THAT ALL THREE REENTRANT CORNER SITES ARE ALLOWED TO GROW.

WHILE IN THE GROWTH OF GERMANIUM CRYSTALS TWINS SERVE A CRUCIAL ROLL, THIS DOES NOT SEEM TO BE THE CASE IN THE GROWTH OF SILICON CRYSTALS. TWINED SILICON CRYSTALS HAVE BEEN GROWN FROM THE MELT [REF 10] BUT IT IS NOT CLEAR THAT FAST GROWTH IS ACHIEVED AS IN THE CASE OF GERMANIUM.

IN OTHER METALS AND INTERMETALLICS TWINS SERVE AS A PREFERRED SITE FOR THE GROWTH OF DENDRITES. ONE EXAMPLE IS THE GROWTH OF CADMIUM CRYSTALS [REF 11]. HOWEVER, WE WILL NOT DISCUSS IT AND LIMIT OURSELVES TO CRYSTALS WITH DIAMOND CUBIC STRUCTURE.

THE KEY TO UNDERSTANDING THE ROLL OF TWINING IN THE GROWTH OF DIAMOND CRYSTALS IN THE CVD PROCESS IS THE STABILITY OF A CARBON ATOM ATTACHED TO THE SURFACE OF A GROWING CRYSTAL AT A GIVEN POINT. DURING THE FORMATION OF NATURAL DIAMONDS (SEE FOR EXAMPLE REF. 12) OR IN COMMERCIAL PROCESSES IN WHICH THE PRESSURE-TEMPERATURE ARE SUCH THAT THE DIAMOND PHASE IS STABLE, GROWTH IS NOT PERFORMED IN A REACTIVE ENVIRONMENT AS IN THE CASE OF CVD GROWTH. A CARBON ATOM ATTACHED TO A FREE SURFACE OF THE GROWING CRYSTALS UNDER EQUILIBRIUM GROWTH CONDITIONS IS STABLE AND CAN STAY IN POSITION THUS FORMING A PREFERRED SITE FOR THE FORMATION OF A NEW {111} PLANE. IN CVD DIAMOND GROWTH SUCH AN ATOM WILL BE LIKELY TO ETCH AWAY BY THE ATOMIC HYDROGEN. CARBON ATOMS WHICH POSITION THEMSELVES IN A REENTRANT ANGLE SITE WILL BE MORE STABLE. WHEN A NEW PLANE FORMS AT THE REENTRANT SITE IT CAN GROW FAST SINCE THERE ARE STABLE POSITIONS AT THE STEP SITE OF THE PROPAGATING PLANE. THE REENTRANT SITE BETWEEN TWINS CAN SHOOT OUT PROPAGATING PLANES AT A FAST RATE WHICH CONTROLS THE LOCAL GROWTH RATE (FIGURE 8).
THE LOCAL GROWTH IS ON ADJACENT {111} PLANES BUT ON A LARGER SCALE THE CRYSTAL GROWS IN THE <211> DIRECTIONS.
THE BEST DEMONSTRATION OF THE EFFECT OF TWINING ON GROWTH IN DIAMOND CUBIC STRUCTURE IS GIVEN BY HAMILTON AND SEIDENSTICKER IN REF. 7.

A CLEAR EXAMPLE OF THE HAMILTON SEIDENSTICKER MODEL CAN BE SEEN IN FIGURE 3. THE PART OF THE CRYSTAL MARKED T1-T2-T1 IS THE SHAPE THAT CAN GROW IN A LATH FORM INDEFINITELY. TO OBTAIN FAST GROWTH IN OTHER DIRECTIONS MORE TWINS ON OTHER {111} PLANES ARE NEEDED. THE GROWTH DIRECTION IN THIS PART OF THE CRYSTAL IS MARKED. THE NEW (111) PLANE WILL START TO FORM IN POINT [A] AND THE GROWTH CONTINUES IN BOTH DIRECTIONS AS MARKED BY THE SMALL ARROWS ALONG THE {111} PLANES.

PRIOR TO THIS POINT A MISFIT BOUNDARY HAS FORMED AT [B]. THE FORMATION OF SUCH A MISFIT BOUNDARY WAS DISCUSSED EARLIER IN THIS REPORT AND THE "V" SHAPE WITH AN ANGLE OF 70.5° BETWEEN {111} PLANES INDICATES THE GROWTH DIRECTION AT THAT POINT (THE CRYSTAL GROWS FROM THE TIP OF THE "V" UPWARD). IT THIS VICINITY ANOTHER "V" SHAPED TWIN COUPLE IS MARKED BY [C].

THE MISFIT BOUNDARY THAT STARTS AT [B] PROPAGATES FURTHER INTO THE PERIPHERY OF THE CRYSTAL ALONG A PATH MARKED BY * * * *. IT IS IMPORTANT TO NOTE THAT THE MISFIT BOUNDARY PROPAGATES PARTLY ON A NON CRYSTALLINE PATH BUT MAINLY ALONG {111} PLANES. THIS POINT WAS ALSO ILLUSTRATED IN REPORT #2.

THE COMMON AXIS OF 5 FOLD TWINS IS AN IMPORTANT NUCLEATION SITE FOR NEW PLANES ALONG THE GROWING CRYSTAL. THIS IS MANLY DUE TO THE 5 REENTRANT ANGLES AROUND THE CENTER (SEE FIGURE TBD). IT MAY ALSO SERVE THEREFORE AS A SUPERIOR NUCLEATION SITE FOR THE WHOLE CRYSTAL AND MAY EXPLAIN WHY MANY OF THE OBSERVED CVD CRYSTALS ARE ICOSAHEDRAL IN SHAPE SINCE THESE MAY HAVE 30 REENTRANT ANGLE SITES AROUND THEM.

SUMMARY

HIGH RESOLUTION OF PLASMA ASSISTED CVD DIAMOND FILMS WAS PERFORMED. THE FILM IS FINE GRAINED WITH GRAIN SIZE OF THE ORDER OF 0.1 MICRON. SEVERAL FEATURES OF THE MICROSTRUCTURE WERE STUDIED AND THEIR IMPORTANCE TO THE UNDERSTANDING OF THE DIAMOND FILM GROWTH EVALUATED.

THE OBSERVATIONS INCLUDE:

1. TWINNING DENSITY RISES AS A FUNCTION OF THE DISTANCE FROM THE CENTER OF THE CRYSTAL.

2. THE TWINS HAVE AN IMPORTANT ROLL IN THE RAPID GROWTH OF THIS KIND OF FILM. THE REENTRANCE ANGLE BETWEEN PARALLEL TWINS SERVES AS A NUCLEATION SITE FOR THE GROWTH OF NEW (111) PLANES.

3. THE CENTER POINT OF TWIN QUINTUPLET HAS FIVE REENTRANCE ANGLES AND THUS SERVES AS A PREFERRED NUCLEATION SITE FOR NEW PLANES.

4. MISFIT BOUNDARIES, BEING THE LOCUS OF INTERSECTION POINTS OF THE GROWING PLANES ON TWO ADJACENT TWINS CAN SERVE AS AN INDICATOR FOR THE LOCAL CRYSTAL GROWTH DIRECTION. THE MAIN SOURCE OF GROWING PLANES CAN THUS BE TRACED BACK TO A QUINTUPLET TWIN POINT.

LIST OF REFERENCES

1. S. IIJIMA, JPN. J. APPL. PHYS. 26, 357(1987).

2. S. IIJIMA, JPN. J. APPL. PHIS. 26, 365(1987)

3. B.E WILLIAMS, H.S.KONG AND J.T. GLASS, J. MATER. RES. 5, 801 (1990).

4. U. DAHMEN, C.J. HETHERINGTON, P. PIRUZ AND K.H. WESTMACOTT, SCRIPTA MET. 23, 269 (1989).

5. R.S. WAGNER, ACTA MET. 8, 57(1958)

6. A.I.BENNETT AND R.L.LONGINI, PHYS. REV. 116, 53(1959)

7. D.R. HAMILTON AND R.G. SEIDENSTICKER, J. APPL. PHYS. 31, 1165(1960)

8. D.R. HAMILTON AND R.G. SEIDENSTICKER, J. APPL. PHYS. 34, 1450(1963)

9. R.G. SEIDENSTICKER AND D.R. HAMILTON, J. APPL. PHYS. 34, 3113(1963)

10. T. ABE, J. CRYSTAL GROWTH 24/25, 463(1974)

11. P.B. PRICE, PHIL. MAG. SER.8 VOL. 4, 1229(1960)

12. A.R. LANG J. CRYSTAL GROWTH 24/25, 108(1974)

APPENDIX 1

DIAMOND DEPOSITION RECORDS

DIAMOND DEPOSITION RECORD

Tube Furnace (A)	Bell Jar (B)	Microwave (C)
Specimen Number 321 C LR-1	Start 4-29-90 3:25	End - 10

Substrate Material 3/4" Si square indented on bottom
surface treatment and dry polished

substrate temperature 650° C

Filament Number	Filament run number
Material	Current
No. of turns	Voltage
Wire diameter	Resistance
Turn diameter	Power
Temperature	Run time

Forward power 1000 W	Top stub 252
Reflected power 0	Substrate position 30
End stub 250	Run time 45 min

Gas mixture

H_2 set point 260	Base pressure
CH_4 set point 1.3	Run pressure
set point	Flow (SCCM)

Comments Free-standing continuous film
as thin as possible

DIAMOND DEPOSITION RECORD

Tube Furnace (A)	Bell Jar (B)	Microwave (C)
Specimen Number 322C LR-2	Start 6-30-90 9:40	End 11:40

Substrate Material surface treatment 3/4" Si square indented on bottom and dry polished

substrate temperature 650°C

Filament Number	Filament run number
Material	Current
No. of turns	Voltage
Wire diameter	Resistance
Turn diameter	Power
Temperature	Run time

Forward power 1000 W	Top stub 252
Reflected power 0	Substrate position 30
End stub 250	Run time 2 hours

Gas mixture

H_2 set point 260	Base pressure
CH_4 set point 1.3	Run pressure
set point	Flow (SCCM)

Comments Thicker continuous film (2 hours)

FIGURE CAPTIONS

FIGURE 1. A TWIN QUINTUPLET (MARKED A) CREATES A 7.5° MISFIT BOUNDARY ALONG ONE OF THE TWIN HABIT PLANES (SEE INSERT). THE MISFIT BOUNDARY GEOMETRY AND IMPORTANCE IS DISCUSSED IN THE TEXT.

FIGURE 2. MISFIT BOUNDARIES CAN TERMINATE AT TWIN HABIT PLANES (AS IN THIS EXAMPLE) OR STRETCH TO THE SURFACE OF THE CRYSTAL AS CAN BE SEEN IN FIGURES 4 AND 5.

FIGURE 3. THE MISFIT BOUNDARY AT [B] BETWEEN THE "V" SHAPED T_2 AND T_3 FORMS ON A SURFACE WHICH IS THE LOCUS OF INTERSECTION POINTS OF GROWING PLANES ON TWO ADJACENT NON-MATCHING TWINS. IT THUS INDICATES THE CRYSTAL GROWTH DIRECTION IN THAT VICINITY (MARKED BY A DARK ARROW). THIS MISFIT BOUNDARY PROPAGATES IN A PATH MARKED BY * * * *. ANOTHER "V" SHAPED PAIR OF NON-MATCHING TWINS IS SEEN AT POINT [C]. THE REENTRANCE POINT AT [A] SERVES AS A PREFERRED NUCLEATION SITE FOR A NEW PLANE WHICH GROWS AS INDICATED BY SMALL DARK ARROWS ALONG T_1 AND T_2. AS NEW PLANES ARE ADDED, THE TWINS GROW IN THE DIRECTION MARKED BY HOLLOW ARROWS.

FIGURE 4. MISFIT BOUNDARIES (ALL ARROWS) ARE MORE FREQUENTLY FOUND IN THE PERIPHERY OF THE CRYSTAL. HOLLOW ARROWS INDICATE ALSO THE LOCAL GROWTH DIRECTION OF THE CRYSTAL.

FIGURE 5. GROWTH DIRECTIONS, MARKED BY ARROWS IN THIS CROSS SECTION OF A COMPLETE CRYSTAL CAN AID IN TRACING BACK THE NUCLEATION SITE OF THE GROWING PLANES TO THE TWIN QUINTUPLET MARKED BY A CIRCLE.

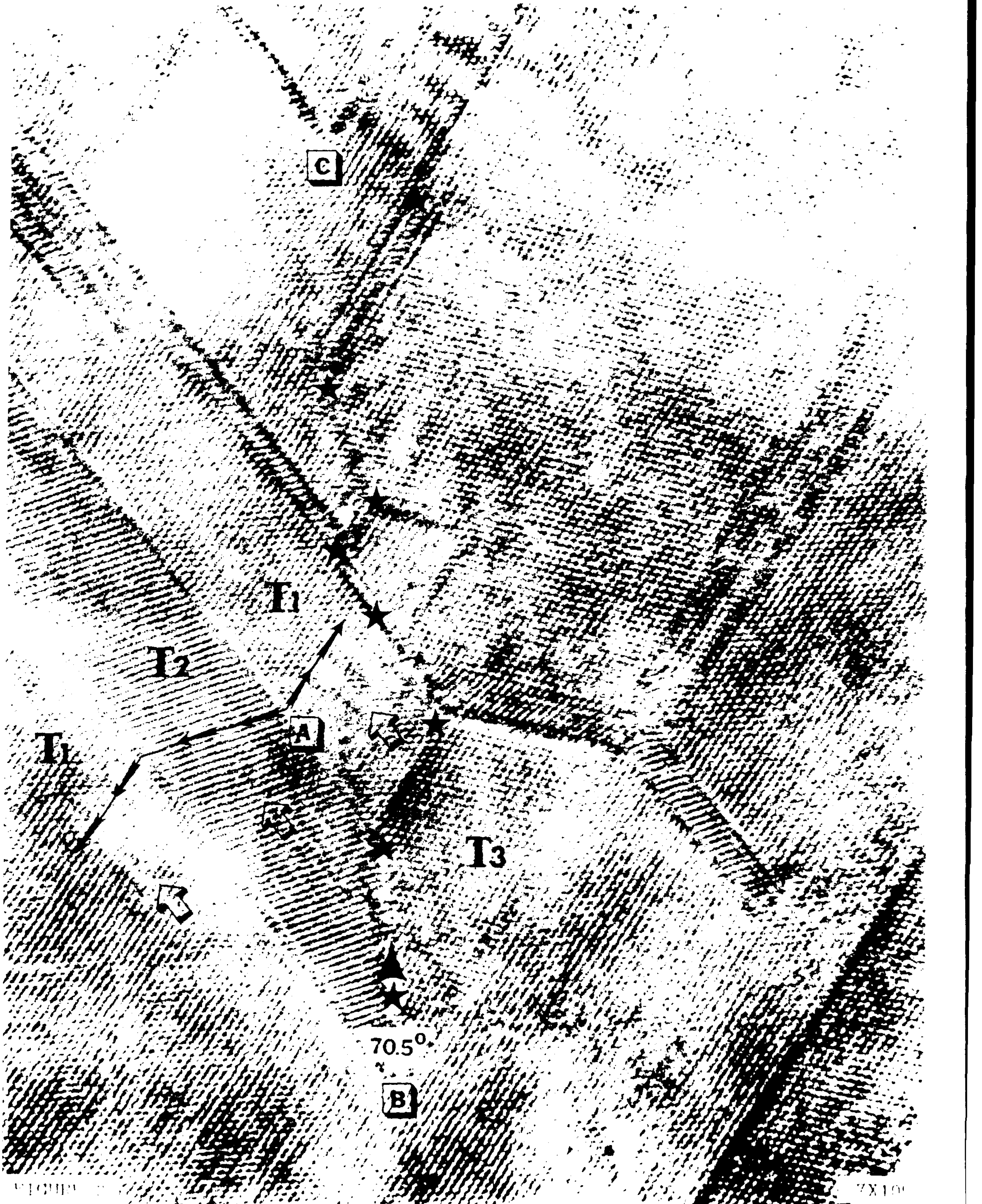
FIGURE 6. TWIN-RICH PARTS OF THE CRYSTAL (MARKED R) GROW FASTER AND THEREFORE PROTRUDE INTO AVAILABLE SPACE WHILE TWIN-LEAN PARTS (MARKED L) LOOSE IN THE COMPETITION TO THE NEIGHBORING CRYSTALS.

FIGURE 7. THE HAMILTON-SEIDENSTICKER [7] MODEL FOR FAST GROWTH OF GERMANIUM CRYSTALS CALLS FOR A SET OF TWO PARALLEL {111} TWINS WHICH FORM 6 REENTRANCE SITES. WHEN 3 OF THE SITES CLOSE UP TO A TIP THE OTHER 3 OPEN UP TO ALLOW FOR CONTINUING GROWTH. THE PREFERED GROWTH DIRECTION BELONGS THUS TO THE <211> FAMILY.

FIGURE 8. THE GROWTH OF A (111) PLANE STARTS AT POINTS ALONG THE 141° REENTRANCE LINE. THE GROWTH CAN CONTINUE THROUGH THE 219° PROTRUSION.


A

[illegible] BOUNDARIES CAN TERMINATE AT TWIN [illegible]
[illegible] SURFACE [illegible]
[illegible] CAN BE SEEN IN FIGURES 4 AND 5


C
T1
T2
T1
A
T3
70.5°
B

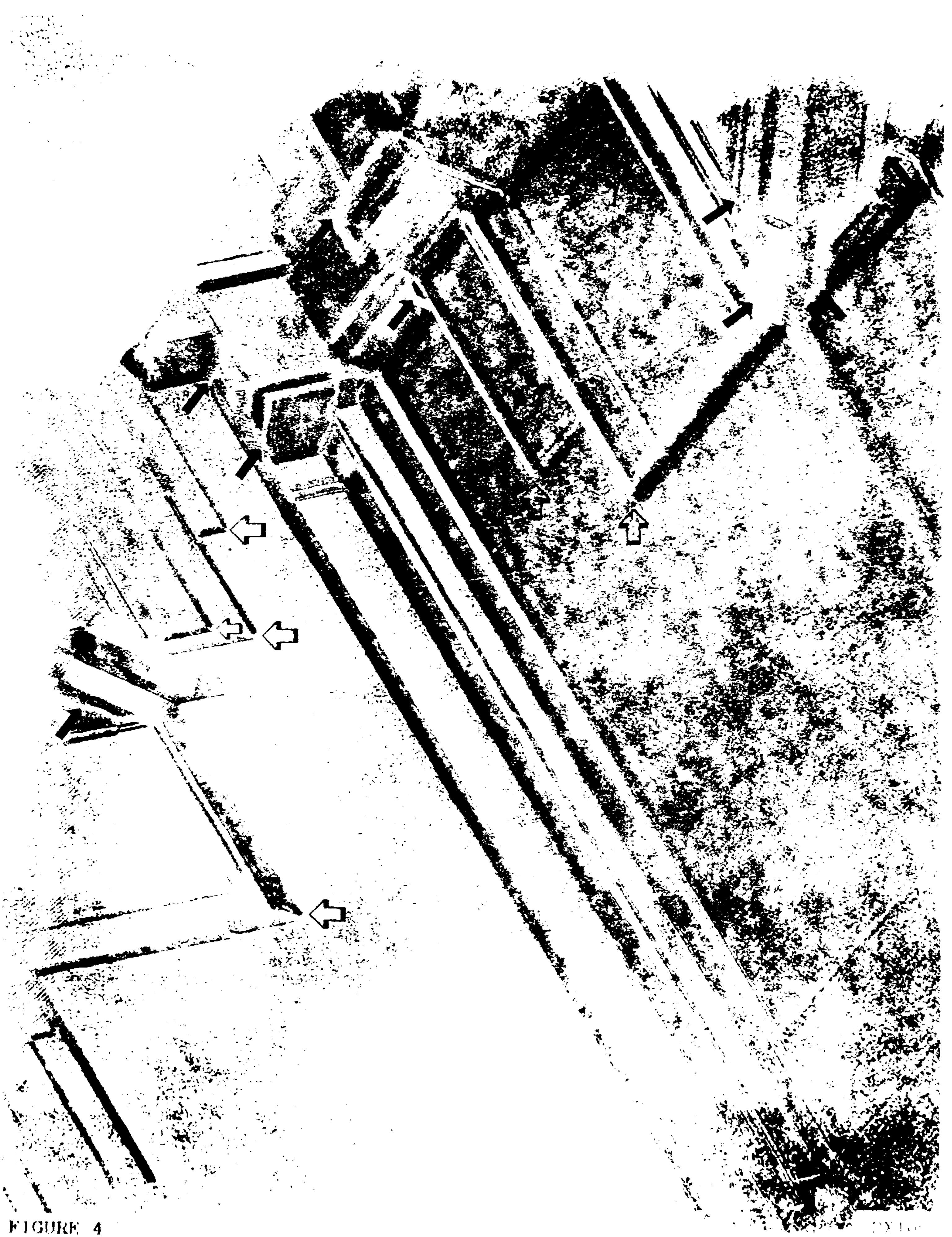


FIGURE 4

FIGURE [illegible]

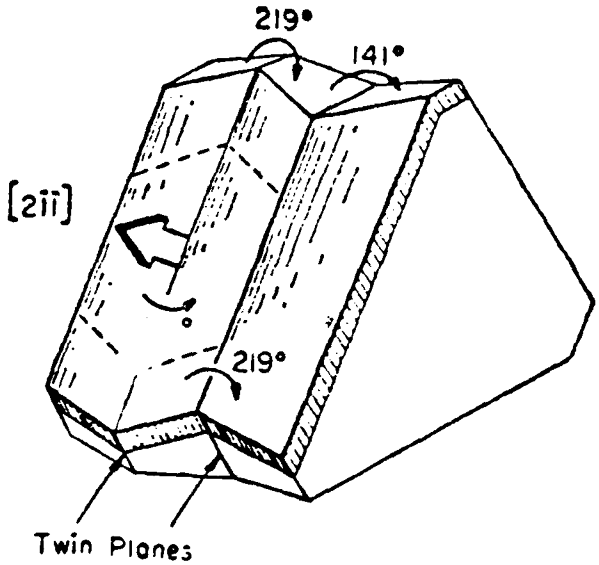


FIGURE 7. THE HAMILTON-SEIDENSTICKER [7] MODEL FOR FAST GROWTH OF GERMANIUM CRYSTALS CALLS FOR A SET OF TWO PARALLEL {111} TWINS WHICH FORM 6 REENTRANCE SITES. WHEN 3 OF THE SITES CLOSE UP TO A TIP THE OTHER 3 OPEN UP TO ALLOW FOR CONTINUING GROWTH. THE PREFERED GROWTH DIRECTION BELONGS THUS TO THE <211> FAMILY.

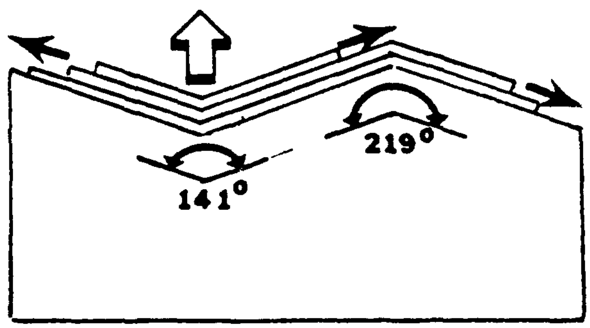


FIGURE 8. THE GROWTH OF A (111) PLANE STARTS AT POINTS ALONG THE 141° REENTRANCE LINE. THE GROWTH CAN CONTINUE THROUGH THE 219° PROTRUSION.

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